Investigating the structure of Splat-Cooled Uranium and Uranium-Molybdenum alloys

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Abstract

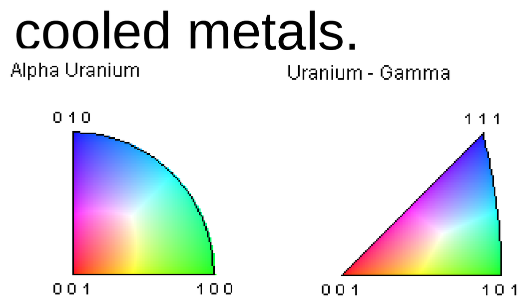
Splat cooled uranium has been used in a range of studies for around 50 years, coming into focus more recently as a source of ‘glassy’ or ‘amorphous’ uranium for super-conductor and electromagnetic experiments. But apart from a very cursory optical metallographic study in the early 60’s[1,2], no physical characterisation work has ever been done.

The range of new techniques which have been developed since the initial study have significantly broadened the possible approaches, and have led to a truly novel study. Perhaps the most significant finding of the study has been the demonstration that using a splat cooling method with cooling rates of the order of 10^6 K/s it has been possible to preserve the high temperature (existing for $T > 775^o$ C) γ-phase at room temperature (RT) in pure uranium, something which was hither-to considered impossible. The γ-phase observed using EBSD were both intra-granular micro-grains (typically siting along sub-boundaries) and inter-granular micro-grains. This correlates with XRD measurements made by the manufacturing research group [N-TH Kim-Ngan *et al*. JdA2012 Friday talk]. Given the relative strength of the XRD signal, it is thought that the γ-phase is concentrated at the outer surfaces of the splat (which, as the point of the most rapid cooling, makes it the most likely position for the preservation of a metastable phase) and the majority of the phase is consequently removed during the electropolishing required to yield a suitable surface for EBSD analysis. As part of the same study, the structure of a range of different splat cooled uranium-molybdenum alloys (4, 11 and 15 at%) has been investigated to determine the stabilising effects of the alloying element. The XRD data shows that the 4 at% Mo sample is a mix of α'+γ, with the 11 at% Mo effectively γ^0 and the 15 at% Mo the expected pure γ-phase.

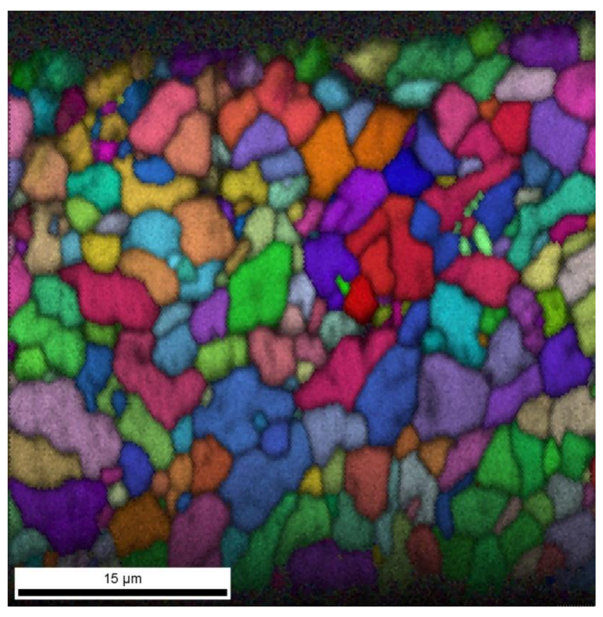
[1] S. Isserow; Early work on rapidly solidified uranium , Journal of Materials Science, 1981,16, 3214-3215; [2] A. R. Kaufmann; Nuclear Metals Inc. (1963); Method and apparatus for making powder, US-3099041

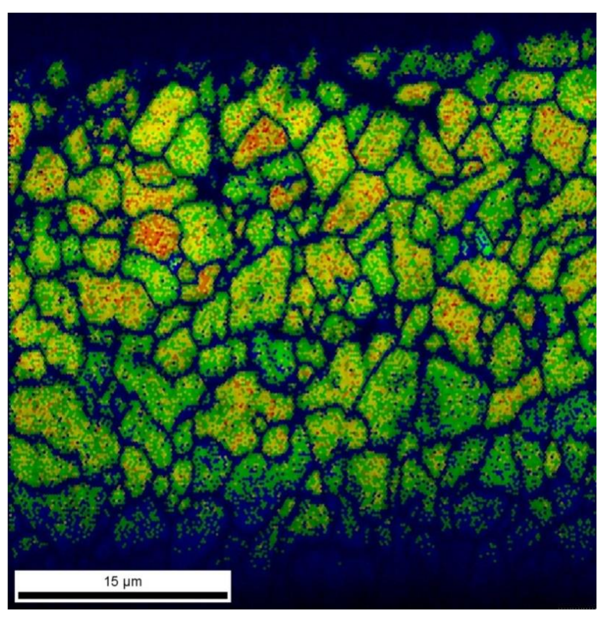
Distorted microstructure – EBSD of cross-sectional cut 0 at% Mo

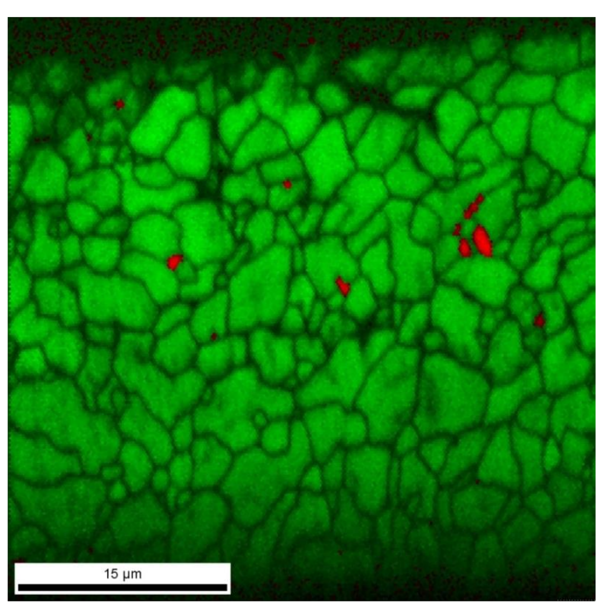
The Inverse Pole Figure EBSD map to the left shows a example EBSD map of a cross-sectional cut through a U-splat. These maps elucidated the unusual microstructure of the splat cooled uranium, which shows a range of distorted grains shapes. Grain sizes were typically large, with the modal average being 24.6 µm diameter, and exhibited a preference for a (101) orientation. A significant number of twins were observed – the most abundant were narrow 69º misorientations ({130} twins), although there were a number of much wider 90º misorientations ({172} twins). No γ-phase was detected with any confidence throughout the cross-section, only α-U (RT phase) was recognized. Despite the distorted structure observed throughout the splat, no evidence for dendritic growth was observed, unlike many other splat cooled metals.



EBSD of surface electropolished 15 at% Mo alloy







An EBSD phase map of the 15 at% Mo alloy confirms the XRD results that the material is γ-U (no detectable α-U). The crystal structure appears to be finer than that of the pure uranium splat (modal average diameter of 3.1 µm, with over 60% of grains between 3.1-5.2 µm), and shows less signs of distortion, in addition to a preference for a (011) orientation. Although one EBSD map indicated that there was a possibility of grain size varying significantly across a cross-section of the sample, this was not supported by other maps recorded in the same region. As such this has been attributed to being an artefact of perspective.

Color Coded Map Type: Phase

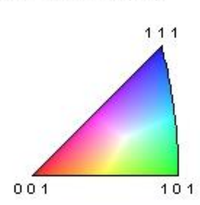
Phase	Total Fraction	Partition Fraction
Uranium Carbide (1/1)	0.024	0.024
Uranium - Gamma	0.976	0.976

Color Coded Map Type: Confidence Index

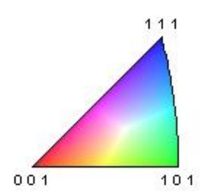
Min	Max	Total Fraction	Partition Fraction
0	0.1892	0.476	0.476
0.1892	0.3784	0.306	0.306
0.3784	0.5676	0.182	0.182
0.5676	0.7568	0.033	0.033
0.7568	0.946	0.003	0.003

Color Coded Map Type: Inverse Pole Figure [001]

Uranium Carbide (1/1)

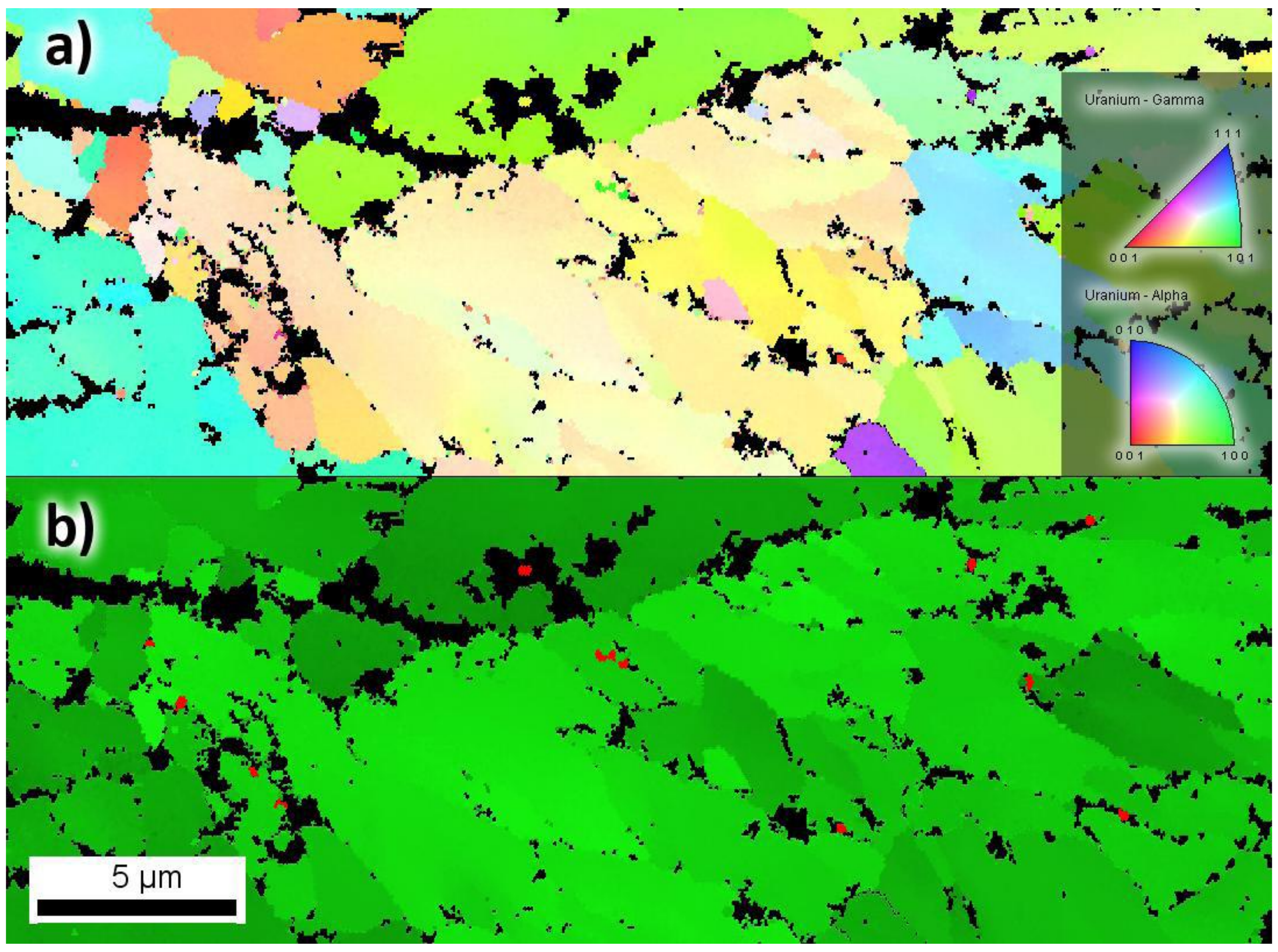


Uranium - Gamma



Preservation of high temperature phases

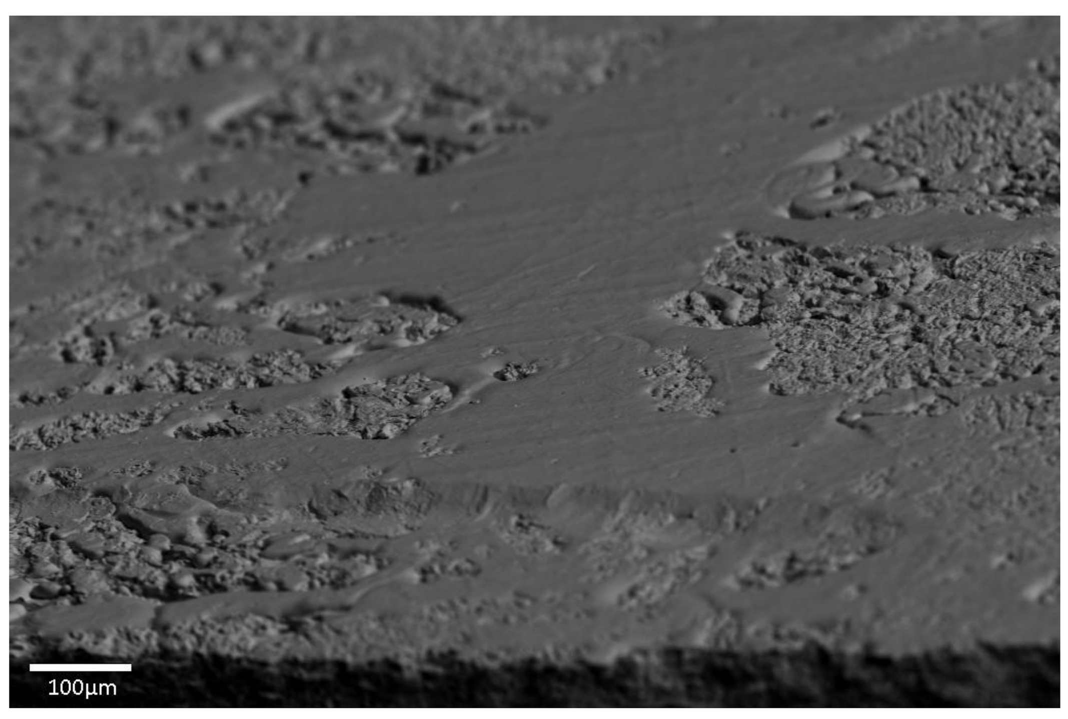
Micro-grains of γ-phase uranium were observed on the sample surface. As the surfaces of the splat would be subject to the most rapid cooling, it is also the region where the γ-phase is most likely to be preserved compared to the bulk of the sample. The scarcity of grains in the EBSD images, does not correlate well with the intensity of the peak in the XRD data – however, as the electropolishing used to prepare the sample for EBSD will have removed a sizable volume of the surface, it is possible that quantities of γ-phase were removed at that point.



Color Coded Map Type: Phase

Phase	Total Fraction	Partition Fraction
Uranium - Gamma	0.057	0.057
Alpha Uranium	0.943	0.943

Figure to the right shows an example SEM image of the surface of a 0 at% Mo sample. The unusual topography was removed during electropolishing, and the subsequent flattened regions have been shown to have no difference in composition to the rest of the material.



Conclusions

It has been shown that the uranium high temperature γ phase can be preserved in pure uranium in small quantities by splat cooling. The γ-U has been observed as both inter -and intra-granular micro-grains, and is thought to concentrate at the splat surfaces. γ phase has also been preserved through alloying with Mo, with only the γ- phase observed in the 15 at% Mo alloy splat.